AMENDMENT NO. 2 FEBRUARY 2018
TO
IS 6932 (PART 2) : 1973 METHODS OF TESTS FOR BUILDING LIMES

PART 2 DETERMINATION OF CARBON DIOXIDE CONTENT

(*Page* 4, *clause* **3.3**) — Insert the following new clause at the end:

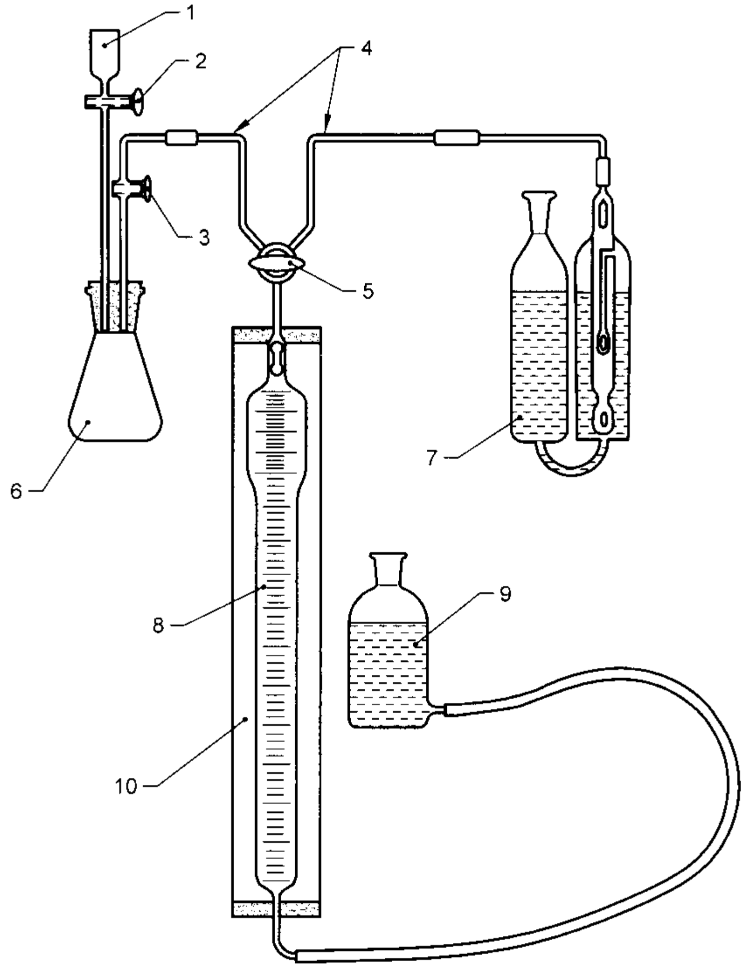
'4 VOLUMETRIC DETERMINATION OF CARBON DIOXIDE (ALTERNATE METHOD)

4.1 Principle

The carbon dioxide contained in the lime in the form of carbonates is given off by reaction with hydrochloric acid and determined volumetrically.

4.2 Apparatus

The apparatus shall be as shown in Fig. 2.



1 DROPPING FUNNEL
2 STOPCOCK 1
3 STOPCOCK 2
4 CAPILLARY TUBES
5 THREE-WAY TAP
6 DECOMPOSITION FLASK WITH A VOLUME OF 50 ml
7 ABSORPTION VESSEL CONTAINING POTASSIUM HYDROXIDE SOLUTION
8 BURETTE
9 LEVELLING VESSEL WITH SEALING LIQUID
10 JACKETED TUBE FILLED WITH WATER

FIG. 2 KLEINE APPARATUS FOR DETERMINATION OF CARBON DIOXIDE

Price Group 2

4.3 Reagents

a) Copper sulphate, $CuSO_4$.

b) Concentrated hydrochloric acid, HCl.
(ρ = 1.18 to 1.19 g/$cm^3$and conforming to IS 265:1993 'Hydrochloric acid — Specification').

c) Dilute hydrochloric acid, 1:1 (1 part of concentrated hydrochloric acid and 1 part of water by volume).

d) Methyl red solution, dissolve 0.2 g of methyl red in water and make up to 100 ml.

e) Sealing liquid, add 20g of sodium sulphate and 5ml of concentrated sulphuric acid to distilled water and make up to 100ml and colour with a few drops of methyl red solution. The sealing liquid shall be saturated with CO_2.

f) Potassium hydroxide solution, containing 50 percent of mass of KOH.

g) Calcium carbonate, $CaCO_3$ dried to constant mass at 250 °C.

4.4 Procedure

The mass of lime sample used depends on the expected CO_2 content and shall be chosen approximately as follows:

a) 2.0 g for 0 percent $\leq CO_2 \leq$ 2 percent;
b) 1.0 g for 2 percent $< CO_2 \leq$ 5 percent;
c) 0.5 g for 5 percent $< CO_2 \leq$ 10 percent;
d) 0.3 g for 10 percent $< CO_2 \leq$ 15 percent; and
e) 0.1g for 40 percent $\leq CO_2 \leq$ 50 percent.

Weigh the sample accurately to 0.1 mg into the decomposition flask and suspend in a little water.

Connect the flask to the apparatus with the aid of the double-bored stopper. A funnel and the feed tube to the measuring burette pass through the stopper. Open the stopcocks in these two lines. Bring the three-way tap to the position such that it connects the flask and measuring burette with one another. Fill the burette with sealing liquid up to the three-way tap by raising the levelling bottle. Now close stopcock 1 and fill the funnel with dilute hydrochloric acid (1:1). Add dilute hydrochloric acid (1:1) to the flask through the dropping funnel until the flask is half full. Stopcock 1 being closed, a little acid remains as sealing liquid in the funnel.

Allow the mixture to react for a few minutes in the cold and then heat it to the boiling point and boil for about another 3 min. Fill the flask completely with dilute hydrochloric acid (1:1) up to stopcock 2 with the aid of the dropping funnel in order to transfer the remaining gas mixture into the burette. Take care that no dilute hydrochloric acid also flows over. Close the burette with the three-way tap. After about 5 min, bring the sealing liquid in the burette and in the levelling bottle to the same level and read off the gas volume V_1.

Turn the three-way tap to connect the measuring burette with the absorption vessel and wash out the air/CO_2 mixture collected. For this, raise the levelling bottle so that all the gas is forced through the potassium hydroxide solution in the absorption vessel. The CO_2 is thereby absorbed. Repeat the absorption operation about seven or eight times until, finally, the measuring burette contains only the residual gas. Close the three-way tap, bring the sealing liquid in the burette and in the levelling vessel to the same level and read off the volume V_2.

The difference in volume (V_1- V_2) corresponds to the carbon dioxide content of the sample.

4.5 Evaluation

Calculate the carbon dioxide content as the content by mass of CO_2, in percent, from the following equation:

$$C = 0.053\, F_1 \frac{(V_1 - V_2) \times P}{T \times m_1}$$

where

C = carbon dioxide content by mass, in percent;
F_1 = correction factor in accordance with **4.6**;
V_1 = volume of the gas before absorption, in ml;
V_2 = volume of the gas after absorption, in ml;
P = corrected barometer reading, in Pa × 100;
T = measurement temperature, in K; and
m_1 = mass of the sample, in g.

If the calibration and determination are carried out directly after one another, the temperature and atmospheric pressure need not be taken into consideration. For this case, the equation is simplified to:

$$C = \frac{4.397\ (V_1 - V_2)\ \text{of measurement}}{(V_3 - V_4)\ \text{of calibration} \times m_1}$$

If 0.100g of $CaCO_3$ is weighed out for the calibration.

4.6 Calibration of the Apparatus

Weigh 0.1000 g of calcium carbonate, dried to constant mass at 250 °C, to an accuracy of 0.1mg into the decomposition flask. Carry out the determination as described in **4.4**. The volume difference (V_3 − V_4) corresponds to the carbon dioxide content of the calibration material. Calculate the correction factor, F_1 of the absorption apparatus from the following relationship:

$$F_1 = \frac{82.96 \times \mathrm{T}}{(V_3\ \text{-}\ V_4) \times \mathrm{P}}$$

The meanings of the symbols correspond to those given in **4.5**. The factor shall be in the range 1.00 to 1.04, otherwise the apparatus is to be checked for tightness and proper functioning and the calibration repeated.'

(CED 04)

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